AECL-9186

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L'ÉNERGIE ATOMIQUE DU CANADA LIMITÉE

COMPUTER ASSISTED TOMOGRAPHY FOR THE NON-DESTRUCTIVE EVALUATION OF HYDROGEN-INDUCED CRACKING IN STEEL

Tomographie informatisée pour l'évaluation non destructive de la fissuration dans l'acier sous l'effet de l'hydrogène

R.L. TAPPING and B.D. SAWICKA

Chalk River Nuclear Laboratories

Laboratoires nucléaires de Chalk River

Chalk River, Ontario

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R.L. Tapping* and B.D. Sawicka

*System Chemistry and Materials Branch

Radiation Engineering Branch
Chalk River Nuclear Laboratories
Chalk River, Ontario, Canada K0J 1J0

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TOMOGRAPHIE INFORMATISÉE POUR L'ÉVALUATION NON DESTRUCTIVE DE LA FISSURATION DANS L'ACIER SOUS L'EFFET DE L'HYDROGÈNE

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RÉSUMÉ

La tomographie informatisée (CAT) a servi à évaluer la fissuration dans l'acier sous l'effet de l'hydrogène, l'acier étant exposé à un milieu saturé de H_2S ("acide"). Dans ce cas, le milieu était celui de l'essai NACE TM-02-84 quant à la susceptibilité à la fissuration sous l'effet de l'hydrogène. On a montré dans une communication antérieure la possibilité d'utilisation CAT dans ce cas. L'étude permet d'étendre l'application CAT à une évaluation de la fissuration. On détermine les paramètres optimaux de la formation d'images par CAT ainsi que les avantages de l'utilisation CAT à la place des techniques d'examen traditionnelles.

* Département de la Chimie et matériaux de systèmes

Département d'Études des rayonnements
Laboratoires Nucléaires de Chalk River
Chalk River, Ontario, Canada KOJ 1JO

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ABSTRACT

Computer assisted tomography (CAT) was used to assess hydrogen-induced cracking in steel exposed to an H_2S-saturated ("sour") environment. In this case the environment was the NACE TM-02-84 test for susceptibility to hydrogen-induced cracking. The feasibility of using CAT in this application was shown in a previous paper. This study extends the application of CAT to a quantitative assessment of the cracking. Optimal parameters for CAT imaging in such an application are determined and the advantages of using CAT in comparison to traditional inspection methods are discussed.

*System Chemistry and Materials Branch

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1. INTRODUCTION

Carbon steels exposed to H_2S-contaminated environments (termed "sour" by the oil and gas industry) frequently suffer various forms of hydrogen damage, one of which is hydrogen-induced cracking, HIC. Hydrogen blistering results from similar causes. It is generally accepted that hydrogen-related corrosion of steels results when hydrogen atoms diffuse into the steel and are trapped in the bulk of the alloy, recombining to form hydrogen molecules and eventually concentrating sufficiently to overcome the local yield stress of the material and hence cause cracking or blistering. The source of hydrogen atoms in a sour environment is the corrosion reaction:

$$Fe + H_2S \rightarrow FeS + 2H^+$$

In order to avoid accumulation of hydrogen atoms, and subsequent recombination to form hydrogen gas, the steel used must be relatively free of internal inclusions; in other words it must be clean.

Inspection of steel before use in a sour environment would, in principle, allow an estimate to be made of the susceptibility of the steel to hydrogen cracking by evaluating the number of inclusions (sulphide, alumina, etc.). At the present time, however, the spatial resolution of routinely-available non-destructive testing (NDT) methods is usually insufficient to properly or reliably identify such defects. Hydrogen cracking in the vicinity of such defects enlarges the defects and provides a distinct interface (gas-solid rather than solid-solid) for investigation by NDT methods. It has been demonstrated in these laboratories that both ultrasonic (1) and γ tomography (2) methods can identify hydrogen-cracked regions in carbon steel. The defect mapping obtainable with ultrasonic methods, however, is not as readily converted to conventional three-dimensional images of cracks as is computer-assisted γ-ray tomography (CAT). CAT is capable of distinguishing small variations in bulk density. The measurable range of the density variations depends on the CAT parameters used and the extent of the bulk density gradient; usually CAT images are measured with a contrast level of 1-3% which implies that density variations of this order can be measured. The defect imaging of CAT is essentially 1:1 with that obtained by destructive (metallographic) methods, and hence with conventional human visual observations.

Hydrogen-induced cracking susceptibility in steels may be assessed using a standard test, the NACE (National Association of Corrosion Engineers) TM-02-84 test (3). The test involves exposure of the steel to an H_2S-saturated acidified sodium chloride solution for two four-day periods, followed by metallographic examination, which involves counting the cracks observed on a specified number of cross-sectional faces. This is a tedious and time-consuming process, and subject to some uncertainty. The results, expressed in terms of the ratio of total crack length and total crack thickness to the total cross-sectioned area, are not highly reproducible and are difficult to relate to in-service performance. The test does, however, identify steels that are either highly susceptible or not susceptible to HIC in sour environments. It is a question of judgement in intermediate cases whether the test results can be used as a guide to in-service behaviour.

CAT scanning of steels subjected to sour environments, and in particular the NACE test for HIC, provides the same information as that obtained metallographically, but without the need to section the steel samples. The CAT scanner can look for defects in any planar cross-section of the sample, for instance the same planes required by NACE TM-02-84, and using image reconstruction techniques can interpolate between planes to provide a full three-dimensional view of a defect - the closer the planes the more precise this reconstruction will be. CAT has been used to identify HIC in a previous study (2), using a different set of samples than here. That work showed CAT to be a useful technique for assessing the extent of cracking. This study extends the application of CAT to quantitative assessments of cracking. CAT scans were carried out with various density and spatial resolutions in order to evaluate the best parameters required for routine examinations of the samples.

2. EXPERIMENTAL METHOD

The NACE TM-02-84 test was followed exactly, using a sample of carbon steel known to suffer hydrogen-induced cracking in-service in Canadian heavy water plants operated by Atomic Energy of Canada Limited. These heavy water plants use a process requiring circulation of large amounts of H_2S-saturated water and moist air at 2 MPa (300 psi) and 30-130°C through towers constructed of carbon steel. Three samples, labelled B3, B7 and B9, each 100 mm x 20 mm x 17 mm were cut from a section of 122 cm (48") diameter (25 mm wall thickness) heavy water plant piping fabricated from A516 grade 70 carbon steel.

Following the immersion portion of the test the samples were examined using CAT, duplicating the procedure to be used later for metallographic sectioning; i.e., scanning the same nine planes (3 per sample) to be sectioned. The samples were then metallographically sectioned at the same locations and optically examined for cracks. A sample of the same steel, not exposed to the test solution, was found not to contain any evidence of cracking using either method.

CAT scans were made using a first generation (translate-rotate, single detector) scanner with an Ir-192 source.

3. RESULTS AND DISCUSSION

Previously it was found that CAT scans obtained with a spatial resolution of 0.5 and 0.35 mm were able to detect the HIC in various samples but without resolving individual cracks (2). In the present work we measured CT images with various spatial resolutions and density resolutions (contrast) in order to find the best CAT parameters for the optimum visualization of cracked regions, while at the same time trying to keep the CAT scanning time low, which is usually the important criterion for industrial applications. With such a compromise, optimal visualization (using CAT) of the cracked regions was obtained with spatial resolutions of 0.5 mm to 0.25 mm and a contrast of 1-2%.

In Figs. 1-4 comparisons of conventional (metallographic sectioning and optical assessment) and CAT images (measured with spatial resolutions of 0.5 mm and 0.25 mm) are made for three sections. CAT scans at 0.5 mm spatial resolution were carried out for four samples simultaneously and the various images are shown in Fig. 1. Scans at 0.25 mm spatial resolution were made separately for each sample and are shown for three faces in Figs. 2-4 (left

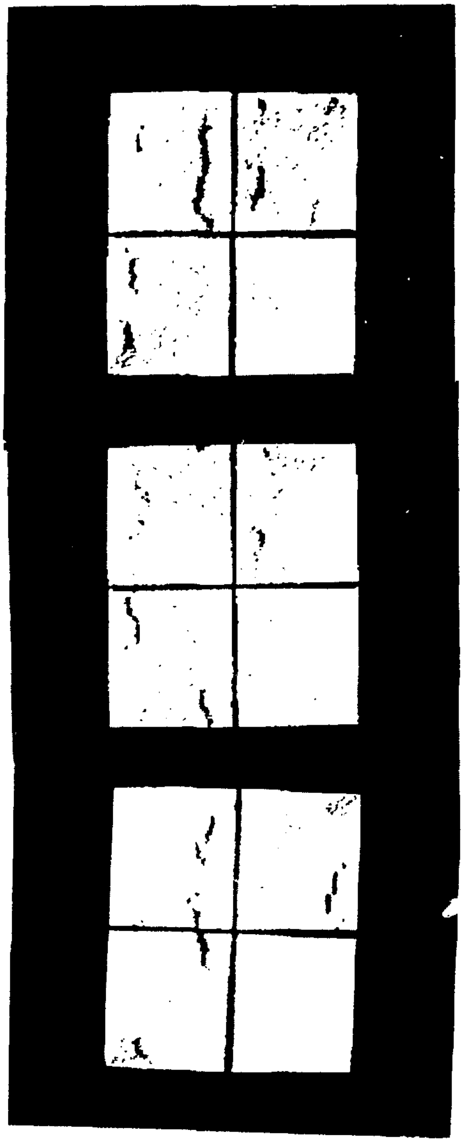

Figure 1: CAT images measured with 0.5 mm spatial resolution and 1% contrast for three "slices" of the four steel samples, B7, B3, B6, B9, clockwise from left upper corner. B6 sample (bottom right) was not exposed to the acid solution and hence contained no cracks.

Figure 2: Comparison of CAT scan (left) and optical, 40x (right) images for the top slice of sample B7. The CAT scan was obtained with a spatial resolution of 0.25 mm and a contrast of 1.4%. The corresponding CAT image obtained with a spatial resolution of 0.5 mm is shown in Fig. 1, top image, where B7 is imaged in the upper left corner of the quartet.

Figure 3: CAT and optical scans for sample B9, top slice. The CAT image has a spatial resolution of 0.25 mm and a contrast of 1.6%. The corresponding CAT scan obtained with a spatial resolution of 0.5 mm, is shown in Fig. 1, top image, where B9 is the lower left sample of the quartet.

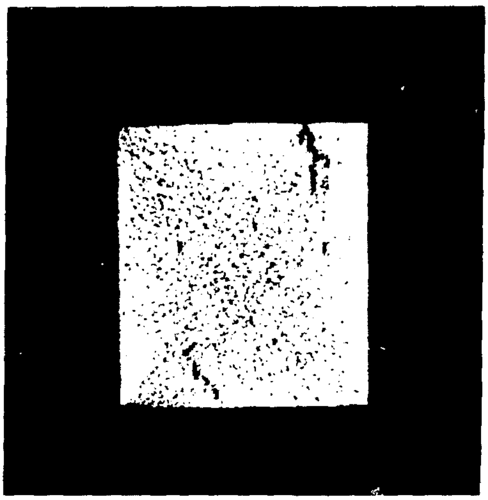

Figure 4: CAT and optical images for sample B9, bottom (third) slice. The CAT image has a spatial resolution of 0.25 mm and a contrast of 1.5%. The corresponding CAT image obtained with a resolution of 0.5 mm is shown in Fig. 1, bottom image, where B9 is the lower left sample of the quartet.

images) together with optical images of the corresponding cross sections (right images).

As may be seen, there is excellent agreement in terms of shape and distribution of the cracks. The optical micrographs present higher resolution images than the CAT images; because sizes smaller than the spatial resolution are reproduced in CAT images in 2-5 pixels, the lengths of cracks are usually represented correctly but the widths of cracks are blurred. It is possible to obtain CAT scans at a higher resolution, at the expense of speed of image acquisition. As will be discussed shortly, this increased resolution is only necessary if an exact duplication of the conventional NACE TM-02-84 practice is required, rather than a rapid and reproducible assessment of HIC. Another reason for the lower resolution of CAT images in comparison to optical images is that CAT averages over a depth of 1-1.5 mm, rather than the micrometre or so in optical microscopy. Because cracks extend some distance through the sample (in fact CAT scans at several closely-spaced planar sections illustrate this very well, see ref. 2) this averaging actually increases the contrast in the cracked regions and thus helps to detect cracks, but it also tends to blur the CAT resolution of the crack size depending on the degree of crack "movement".

A small concern with comparing the visual and CAT images is that the sectioning technique necessarily removes material, in this case approximately 0.5 mm per slice. Thus attempts were made to ensure that all mechanical cuts were such that the resultant polished surface was precisely that scanned in the CAT technique. Usually small discrepancies will not affect the shape and number of cracks intersecting the surface, because, as noted earlier, these cracks extend some distance through the sample. Occasionally, however, the sectioning technique will intersect a crack pattern at a point where it is changing rapidly over a small distance, and in such a situation the CAT and visual images will show discrepancies. As noted earlier, CAT scans represent an average over a finite depth. Thus, CAT can give a much more reliable indication of the degree of cracking than the optical evaluation of NACE TM-02-84, by integrating over a specified depth and exposing cracking that the metallographic/optical procedure may miss.

A major advantage of CAT over metallographic techniques, which has not been addressed in this paper, is the ability of CAT to reconstruct a three-dimensional image of a crack pattern in a sample by interpolation between a series of planar scans. The more closely spaced the planes, and the more scans that are taken, the more accurate the reconstruction. For industrial purposes only a few planes need to be scanned in order to obtain a useful 3-D image of a crack.

The NACE TM-02-84 test procedure requires a quantitative assessment of HIC to be made. The term "crack" will refer now to the cracked region usually composed of a large number of closely spaced small cracks. The HIC assessment is carried out by summing all "crack" (cracked regions) widths and lengths for all sections, and expressing the results in terms of a percentage of examined surface area that is cracked. For instance a crack length ratio, CLR, is obtained by summing all the crack lengths and dividing by the total lengths of sample faces examined. Cracks spaced within 0.5 mm of each other, either transversely or longitudinally, are considered part of the same crack.

This latter criterion is important when considering the comparison between visual and CAT crack assessments.

Table 1 shows some representative data for nine faces, three from each of three samples of the same steel, where only total lengths and widths of the cracked areas are presented. These have not been converted to crack length or thickness ratios, since this would not change the relative numbers given or the discussion to follow (CAT data were obtained from scans with a spatial resolution of either 0.5 mm or 0.25 mm; both give similar results within the accuracy of the CAT measurements which in the two cases is 1 mm and 0.5 mm, respectively). It is immediately apparent from Table 1 that the biggest discrepancy between the CAT and visual lengths and widths is in the width assessments. This is a direct consequence of the CAT resolution and the fact that these values were obtained from the CAT scan photographs simply by using a ruler. The greater resolution of the optical micrographs, plus the fact that the crack dimensions were measured under a microscope, will tend to reduce the crack dimensions relative to CAT. A computerized algorithm to measure crack dimensions directly from the computer data obtained by CAT would eliminate some of this blurring.

The accuracy of the determination of the size of the cracked areas from the CAT images is limited by the spatial resolution used to obtain the image. Crack widths less than the spatial resolution will then have an inherent width in a CAT image of 1-3 times the spatial resolution. The spatial resolution used here gives a good measure of the crack length, but an overestimate of the crack width. For practical purposes, however, this distinction between CAT and visual crack assessment is not important. Before discussing this point further, note that the crack areas, presented here simply as $\Sigma(\ell_i \cdot w_i)$, where ℓ_i and w_i are the lengths and widths of each crack (cracked region), respectively, given in Table 2, show some interesting comparisons. In some cases there is a discrepancy between visual and CAT determinations; in several cases, however, there is excellent agreement between the two. Generally, in diffuse cracks, i.e., those composed of a large number of small cracks, the overall extent of cracking seen by an optical microscope and relatively low-resolution CAT is essentially the same, while for isolated and very narrow cracks the discrepancies become more significant. CAT data which are averaged over a finite depth of the sample are more representative of the whole sample. For example, for slice 2 , sample B3, the surface exposed after mechanical cutting shows no cracks (optical examination) while the CAT scan of the layer having a thickness of 1.5 mm shows two cracked regions (see Figure 1).

Regardless of how the crack dimensions are evaluated, it is known that different samples of steel from the same heat or ingot will often show quite distinctly different test results for HIC sensitivity. This is a result of inhomogeneities and is a practical limit to the HIC test reproducibility. It is also well known that the correlation of in-service performance and HIC tests, including NACE TM-02-84, is not very precise. In fact, experience with heavy water plants where a strong program has existed for testing steels for HIC before service and comparing with in-service behaviour (4), shows that very wide windows of HIC test criteria are required. The test results are readily quantified, but this quantification can only be regarded as an approximate number with very large uncertainty limits.

Coming back, then, to the CAT results of Tables 1 and 2, it seems apparent that the CAT results, while not exactly duplicating the visual ones, are

TABLE 1: Comparison of CAT and visual estimates of crack lengths (ℓ) and widths (w) for each sample face. ℓ and w are the sums of lengths and widths of particular cracked regions, $w=\Sigma w_i$, $\ell=\Delta\ell_i$. All dimensions are in mm.

Sample	Slice Number					
	1		2		3	
	CAT	Visual	CAT	Visual	CAT	Visual
B3	ℓ= 8 w= 4	ℓ=9.5 w=2.5	ℓ= 8 w= 4	ℓ=0 w=0	ℓ= 7 w= 2	ℓ=10.4 w= 3.1
B7	ℓ=18 w= 5	ℓ=16.5 w= 2.9	ℓ= 5 w= 4	ℓ=6.1 w=1.9	ℓ=12 w= 4	ℓ=11.4 w= 3.7
B9	ℓ=12 w= 4	ℓ=10.6 w= 3.6	ℓ=12 w= 4	ℓ=6.8 w=1.9	ℓ= 8 w= 3.5	ℓ= 8.3 w= 2.5

TABLE 2: Comparison of CAT and visual estimates of total cracked area (mm^2), $\Sigma(\ell_i \cdot w_i)$, for each sample face.

Sample	Slice Number					
	1		2		3	
	CAT	Visual	CAT	Visual	CAT	Visual
B3	16	5.6	16	0	14	10.0
B7	48	23.2	11	9.5	28	9.3
B9	18	13.0	33	13.0	14	9.7

undoubtedly at least as reliable as the visual procedures for HIC susceptibility testing. In principle, it would be possible to duplicate the visual results exactly with CAT by using higher spatial resolutions. However, the crack assessment using CAT with low resolution is as good as the optical technique and considerably easier. Overall, spatial resolution here is not an important difference between the two approaches while the examination by CAT of a layer having a limited thickness is an advantage over the optical examination making CAT results highly reproducible within the same sample. The principal advantage of using CAT to evaluate the degree of cracking is the non-destructive nature of the procedure. It is also considerably faster to use CAT. Again, in principle, it is possible to carry out nine total scans of three samples in minutes, including quantitation of results, rather than the days now required when this part of the procedure is carried out manually.

4. CONCLUSIONS

It has been shown that computer-assisted γ-ray tomography (CAT), a non-destructive technique capable of imaging defects in solids by measurement of density gradients, can be used to assess hydrogen-induced cracking (HIC) in steels. The CAT images match 1:1 with the visual images, and quantitative information obtained from those images is directly applicable to the assessment of a particular steel for sour service. An advantage of CAT is that the information can be obtained faster, non-destructively and that it averages over a volume in the steel sample rather than a single plane which makes it more representative of the steel material. If desired, a three-dimensional image of the crack pattern may be obtained.

5. ACKNOWLEDGEMENTS

The authors would like to thank P.A. Lavoie, D.J. Disney, C.J. Allan and P.W. Reynolds for contributing to various phases of these experiments and to the data evaluation.

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